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# FY2014 LX-21 Aging and Compatibility

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# High Explosive Compatibility: Aging and Compatibility of LX-21

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The tasks performed in FY14 to support our overall goals include: 1) Select a mature formulation for future aging and compatibility experiments 2) Perform gross compatibility testing for a number of LX-21/material combinations using three different evaluation techniques (DSC, TGA, and CRT) 3) Evaluate moisture sorption and diffusion characteristics of LX-21 4) Get the infrastructure and procedures in place to begin long-term accelerated aging experiments with LX-21.

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## 1.1 Introduction

The LLNL Chemical Reactivity Test (CRT) was developed at LLNL in the early 1960's [1] and is currently used to assess the short to medium term thermal stability of high explosives (HE) and the chemical compatibility of HE which comes in direct contact with other materials used in explosive operations. The CRT is run at a given constant temperature for a standard amount of time. In the CRT materials of interest are heated in a sealed reaction vessel under controlled conditions and a gas chromatograph (GC) is used to identify and quantify selected gas species which are typically produced by thermal decomposition of HE [1-5]. Results of the test are reported and archived data is made available in the online version of the LLNL Explosives Reference Guide [2]. Over the past four decades, CRT procedures and instrumentation have been updated and refined periodically, other than that not much has changed. While this approach has historical significance and is a suitable method for materials screening it only provides limited information on long-term incompatibilities of materials.

Other analytical tools for rapidly evaluating the compatibility of high explosives include differential scanning calorimetry (DSC) and thermal gravimetric analysis (TGA). While these guidelines are not regularly applied by the DOE to explosives, they are credible metrics for compatibility and are therefore worth evaluation. The DSC method is referenced in a DoD standard (MIL-STD-1751A) and both DSC and TGA are described in the North Atlantic Treaty Organization (NATO) STANAG 4147 that describes several ways to evaluate the compatibility of explosives and other materials.[6-7] There is significant value in taking an integrated approach to compatibility utilizing CRT, DSC, and TGA as complementary screening techniques.

This year, energetic materials were selected for this study based on high priority materials for upcoming programs. More specifically, the explosive 2,6-diamino-3,5-dinitropyrazine-1-oxide (LLM-105) and its PBX formulation with Viton RX-55-CW (94 %wt. LLM-105; 6 %wt. Viton; copper phthalocyanine dye) were exclusively studied. For the past two years there has been a significant effort to develop and certify this formulation as LX-21. This material is a promising candidate for future booster applications in the stockpile due to its combination of desirable performance and good safety characteristics. The two-year effort has produced a very mature form of the formulation. The selected formulation has been optimized in terms of

particle size, additives, and processing methods and is an excellent candidate for detailed and prolonged aging and compatibility efforts. This course of study also provides a good opportunity to evaluate the LLM-105 based formulation with those using TATB newly produced at the BAE Holston plant. This TATB will be the source material for future stockpile use.

The tasks performed in FY14 to support our overall goals include: 1) Select a mature formulation for future aging and compatibility experiments 2) Perform gross compatibility testing for a number of LX-21/material combinations, 3) Evaluate moisture sorption and diffusion characteristics of LX-21 4) Get the infrastructure and procedures in place to begin long-term accelerated aging experiments with LX-21.

## 1.2 Methods

The main goal of this task is to utilize new and existing methods and instrumentation to assess the compatibility and aging behaviors of stockpile relevant HE and HE/materials combinations. New analytical instrumentation and methods now enable a more comprehensive understanding of different aging and compatibility issues. Enhanced Surveillance Campaign (ESC) investments in FY09-12 updated and revitalized our HE compatibility and aging infrastructure, which puts us in position to undertake such a study. In this year's effort, candidate HE and HE/material combinations are evaluated for compatibility using the following techniques.

1. Differential scanning calorimetry (DSC)
2. Chemical Reactivity Test (CRT)
3. Thermal gravimetric analysis (TGA)
4. Sorption/Outgassing analyses

Traditionally the CRT has been used as the benchmark for HE compatibility and even extended to coarse aging predictions, so one can see the potential value of a more comprehensive approach taken here.

Materials to be considered in this study include HE and non-HE materials as well as combinations of said materials. Materials studied this year are tabulated below in Table 1. Our approach involves stockpile relevant materials having their baseline compatibility evaluated using the techniques 1-3 above. This year, materials were selected for this study based on high priority materials for upcoming programs. Specifically the PBX formulation RX-55-CW (94 %wt. LLM-105; 6 %wt. Viton; copper phthalocyanine dye) were exclusively studied.

The non-HE materials are of keen interest due to their historical use, however, over the years commercial and weapon complex supplies and capabilities for these materials have either decreased or ceased all together. In some cases the "old" material is being reproduced after many years of dormancy. This can lead to lack of confidence in the newly produced material through changes in ingredients due to availability or regulations or simply due to a lack of experience in producing it. The lists below summarize both the HE and non-HE materials that are being evaluated under this task plan. Compatibility combinations of materials from each list will be principally studied.

**Table 1.** This table contains a list of selected materials for compatibility screening performed this year.

HE materials	HE lot identification	Non-nuclear materials
LX-17-1	C-063	Halthane 73-18 urethane adhesive
RX-55-CW (94% wt. LLM-105; 6 %wt. Viton) with Copper phthalocyanine blue dye	C-596	FK800
LX-16		APC 2.5 silicone
TATB wet aminated “new”	C-668	Halthane 88-3 urethane adhesive
TATB dry aminated “new”	C-666 (HOL13D297-001)	SE1700 silicone
RX-03-HD	C-666 (92.5%new DA TATB/7.5% new binder FK800)	
RX-03-GX-2 (LX-17-1)	C-668 (92.5%new WA TATB/7.5% new binder FK800)	
LX-16-0	C-518	

Of particular interest here are the results for the “new” TATBs being produced at the BAE Holston Army Ammunition Plant. After nearly three decades the TATB production process is being brought back online by a joint DoD/DOE effort. The TATBs and their subsequent PBXs produced by this revitalization program will be those available for future stockpile applications and thus it is critical that their compatibility with RX-55-CW be established.

To bring this study more in line with the overall materials and compatibility program for future stockpile applications we have incorporated aspects of that plan into our work this year. More specifically, we have focused on executing two tests for materials compatibility: 1) Differential scanning calorimetry (DSC) and thermal gravimetric analysis (TGA). The DSC method has been evaluated over the past two years under this program and looks to be a suitable method for future use at LLNL. New this year is the use of TGA for a compatibility screening test. This is in addition to the chemical reactivity test (CRT) that is already commonly applied throughout the NNSA complex for high explosives. This new approach is also consistent with efforts by colleagues at LANL who are now implementing the DSC test for their HE compatibility portfolio. For more details on the Materials and Compatibility Plan please review ESC reports authored by Elizabeth Glascoe.[8]

### 1.2.1 Baseline compatibility (CRT/DSC/TGA) of relevant LX-21/material combinations

#### *Selection of baseline mature LX-21 source material*

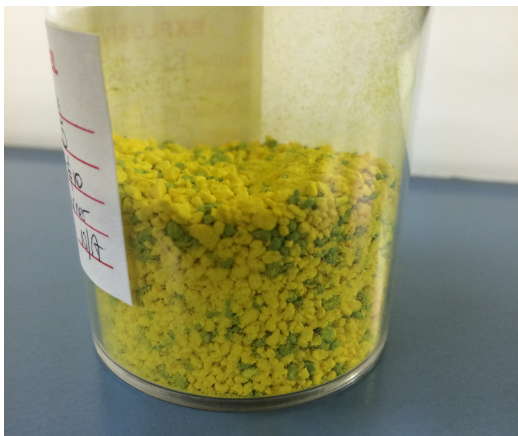
A major development this year was the selection of a mature version of the LX-21 formulation. This version of the booster formulation material has reached a point where its source material, purity, size distribution, and dye are very near if not at their final compositions and concentrations. Once at this point it stands to reason that a extensive compatibility and aging program be enacted.

The major formulation development this year was the selection of an appropriate dye to add to allow visual identification of the explosive. This is a practice that has precedent in the NNSA. To that end, a series of commercial blue dyes were obtained and tested for their compatibility with LX-21 type formulations as well as their ability to color the Viton. Compatibility tests were performed via CRT with the result shown below in Table 2. All of these dyes are compatible with the LX-21, however, some do not color the material well and others lead to extensive foaming during formulation. The dye with the combination of the best (lowest) CRT results, the least foaming on formulation, as well as its formulation retaining its color is the copper phthalocyanine dye. This combination of attributes led to its selection as the dye in LX-21.

**Table 2.** This table contains the results of thermal stability tests for the respective LX-21 formulations that utilized different dyes.

<b>RX-55 Formulation</b>	<b>DYE</b>	<b>CRT Data (cc gas/g HE)</b>
RX-55-CG	Cu Phthalocyanine	0.008
RX-55-CH	Alizarin Blue Black B	0.0616
RX-55-CI	Basic Blue #3	0.0692
RX-55-CJ	Celestine blue	0.0463
RX-55-CK	Indigo	0.0328
RX-55-CL	Methylene Blue	0.2708
RX-55-CM	Mordant Blue	0.1092
RX-55-CN	Basic Blue #41	0.2103
RX-55-CO	PhTh	0.0350

To that end the RX-55-CG formulation batch above was combined with additional un-dyed prill to yield a master batch which for tracking purposes is RX-55-CW. Photos of the mixed prilled RX-55-CW as well as pressed hemispherical part of the formulation are shown below in Figure 1. This formulation was used in nearly all of our micro-compatibility experiments this year and will be used in all of the planned aging studies.



A)



B)

**Figure 1.** Photos of the dyed formulation RX-55-CW and a pressed booster part with a dyed formulation. It is worth noting that the combination of the blue dye and the yellow LLM105 gives a green final color to the composite.

As stated in the Introduction, LLNL has a long history of utilizing the CRT screening method for compatibility. Archived data goes back more than 50 years and provides ample opportunities to compare a large amount of data to results from new materials. While there is extensive data on many HE/materials combinations in the database, new materials or those produced by new or re-instated commercial methods have not been evaluated by CRT. One important outcome of this year's effort was to obtain baseline CRT on these LX-21/materials combinations. Table 3 contains a summary of a subset of these material combinations and tracks the relative progress of CRT analyses.

**Table 3.** Baseline CRT compatibility matrix for HE materials performed in FY2014 are shown below.

	Lot#	RX-55-CW	TATB (new: DA)	TATB (new: WA)	LX-17-1 (legacy)	RX-03-HD (new LX-17-0)	RX-03-GX-2 (new LX-17-1)	LX-16	APC 2.5 (cured)	Halthane 73-18 (cured)	Halthane 88-3 (cured)	FK800 binder	SE1700 silicone
RX-55-CW	C-596	0.01	0.01	0.03	0.02	0.02	0.07	0.56	0.09	0.07	0.07	0.01	0.02
TATB (new: DA)	C-666		NA										
TATB (new: WA)	C-668			NA									
LX-17-1 (legacy)	C-063				0.12								
RX-03-HD (new LX-17-0)	C-666/FK800					NA							
RX-03-GX-2 (new LX-17-1)	C-668/FK800						NA						
LX-16	C-518							NA					
APC 2.5 (cured)	#13-112								0.06				
Halthane 73-18 (cured)										0.78			
Halthane 88-3 (cured)											0.49		
FK800 binder	#1											0.23	
SE1700 silicone													0.05
Pass													
Fail													
Not Completed													
Thermal stability													
Results in cm <sup>3</sup> gas/g of explosive													

Table 3 contains a summary of the results from the baseline CRT compatibility matrix for HE/materials combinations. The results indicate acceptable levels of chemical interaction between all studied material combinations. Briefly, an acceptable level of chemical interaction is defined to be a 1:1 (by mass) combination of the two materials that evolved less than 1.5 cm<sup>3</sup> gas/gram of HE after 22 hours at either 120°C or 80°C. For reference 0.75 cm<sup>3</sup> gas/gram is considered minor, however, acceptable reaction between components. The full CRT method is described in more detail in references [1-4]. While inspection of Table 3 shows all combinations as acceptable what is possibly more revealing is the magnitude of the CRT results obtained. As one can see the matrix above is not complete so this report constitutes a work in progress. Additional notes to be made include the study of SE 1700 silicone from Dow Corning. This material has been shown to be amenable to advanced manufacturing techniques and is being intensely studied in the Additive Manufacturing Initiative at LLNL. A review of the raw data shows that none of these combinations produced anywhere near the minor reactivity level. While hardly comprehensive, this set of data suggests that LX-21 is particularly stable in the presence of these materials.

In an attempt to correlate historical CRT compatibility data with that from other analytical techniques an identical campaign was started in FY2012 to study compatibility using differential scanning calorimetry (DSC).[9] This campaign was continued with the FY2014 work reported here. The HE/material combinations studied in FY2014 by this approach are summarized below in Table 4. One will notice a concerted effort to study the same HE/materials pairs as was evaluated by CRT in Table 3. This was done to allow the comparison of results from both methods, which will enable both the pros and cons of both approaches to be more clearly evaluated.



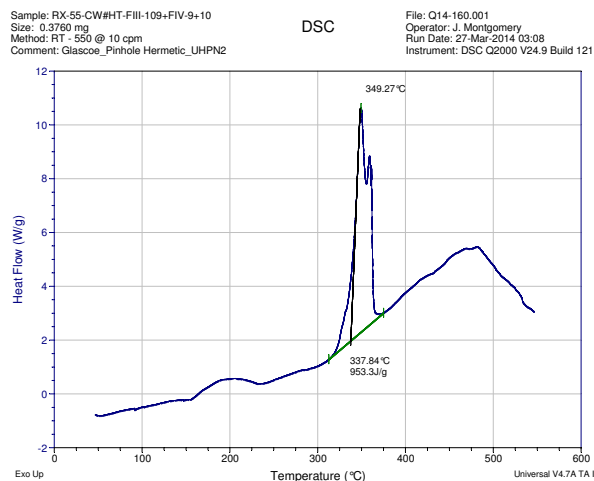
**Table 4.** The baseline DSC compatibility matrix for HE materials performed in FY2014 is shown below.

HE/DSC	Lot#	RX-55-CW	TATB (new: DA)	TATB (new: WA)	LX-17-1 (legacy)	RX-03-HD (new LX-17-0)	RX-03-GX-2 (new LX-17-1)	LX-16	APC 2.5 (cured)	Halthane 73-18 (cured)	Halthane 88-3 (cured)	FK800 binder	SE1700 silicone
RX-55-CW	C-596	350	2*	1*	2*	4*	4*	4*	2*	55*	56*	1*	
TATB (new: DA)	C-666		386										
TATB (new: WA)	C-668			386									
LX-17-1 (legacy)	C-063				385								
RX-03-HD (new LX-17-0)	C-666/FK800					385							
RX-03-GX-2 (new LX-17-1)	C-668/FK800						384						
LX-16	C-518							206					
APC 2.5 (cured)									353**				
Halthane 73-18 (cured)										NA			
Halthane 88-3 (cured)											399**		
FK800 binder	#1											453	
SE1700 silicone													NA
Pass													
Fail													
Not Completed													
Decomposition peak Temperature of neat material (°C)	* Shift in lowest energetic peak (°C)	** determined by SDT											

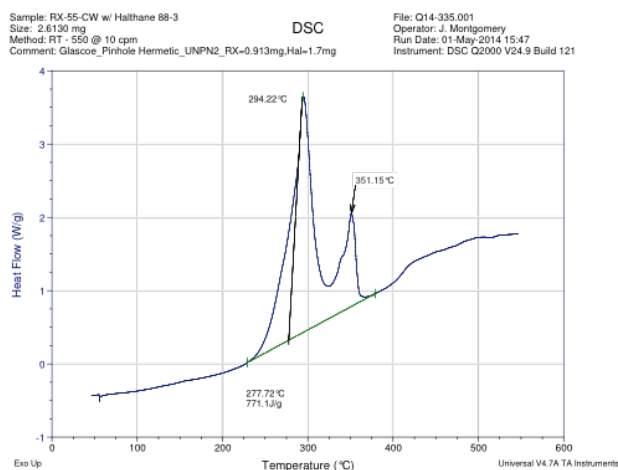
It is important to note that while the STANAG 4147 document calls for DSC and TGA analyses to occur at 2°C/minute the results presented here are from analyses at 10°C/minute. A series of comparison runs at both scan rates were performed and analysis of the data shows no discernable difference in the results. Therefore in the interests of efficiency the 10 degree per minute runs are used to minimize instrument time. This is the heating rate described in MIL-STD-1751A. The subsequent thermogram is evaluated by comparison with those of the neat HE and alien materials respectively. Acceptable compatibility is confirmed if there is less than a 4°C depression in the temperature of the peak exotherm. If the peak temperature shift is > 4°C but less than 20°C the pair is considered borderline compatible and if the shift is > 20°C the pair is considered incompatible. While this approach has not been regularly utilized by the DOE, it is commonly employed by the other agencies (DoD and NATO) and is another metric for compatibility and is worth evaluation.

Review of the data in Table 3 indicates that several of the compatibility evaluations with RX-55-CW result in very little to no shift in the DSC exotherm position. Many of these combination pass both the STANAG and MIL standards for DSC compatibility. However it appears that both Halthane formulations (73-18 and 88-3) are incompatible with RX-55-CW by the DSC criteria. For reference, Halthane is a polyurethane adhesive that consists of polyol and isocyanate pre-polymers. It is a material that finds regular use in the complex. A mixture of each adhesive with RX-55-CW results in a shift in the HE exotherm of ~ 55°C to lower temperatures from ~ 350°C to ~ 290°C. As this is a combination worth further investigation the DSC scans for neat RX-55-CW and its mixture with Halthane are shown below in Figure 2.

A)



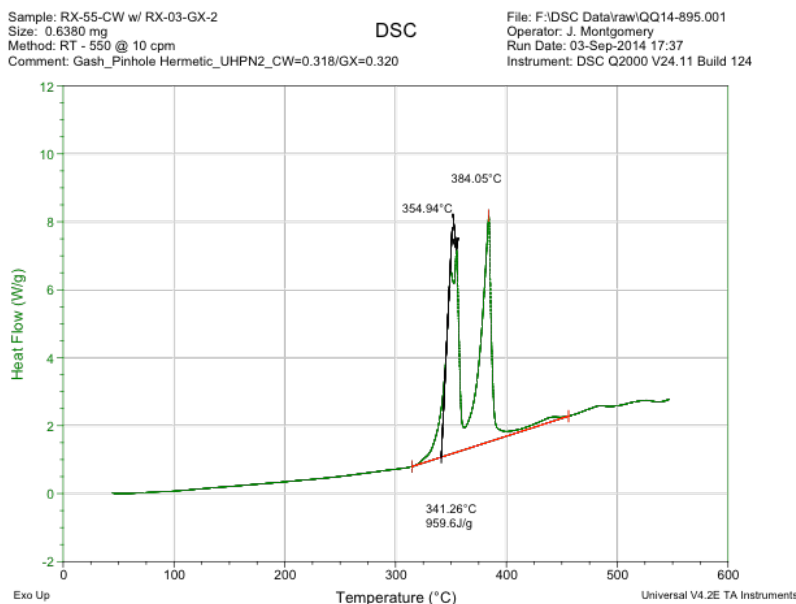
B)



**Figure 2.** DSC compatibility scan of the A) LLM105 formulation RX-55-CW and its B) mixture with the adhesive Halthane 88-3. Note the first peak temperature of decomposition for the explosive formulations has shifted ~55 °C lower than in the base material.

One will notice that the primary exotherm at 349°C in the neat RX-55-CW material has broadened and shifted to 290°C in the mixture with Halthane 88-3. This type of interaction is considered to indicate a significant incompatibility between these materials. It is interesting to note that these combinations pass the CRT. It is perfectly valid to question the importance of a shift in exotherms at a temperature of ~300°C to compatibility. It is known that pyrazine rings react readily with primary amines. It is possible that the Halthane components may have trace primary amines, as determined by Baker et al.[10]

For reference it is instructive to view the DSC scan in Figure 3. This scan is for the mixture of RX-55-CW and the TATB-based explosive RX-03-HD.



**Figure 3.** DSC compatibility scan of the LLM105 formulation RX-55-CW and the TATB-based formulation RX-03-GX-2. Note the peak temperature of decomposition for the two explosive formulations are 354 and 384°C respectively.

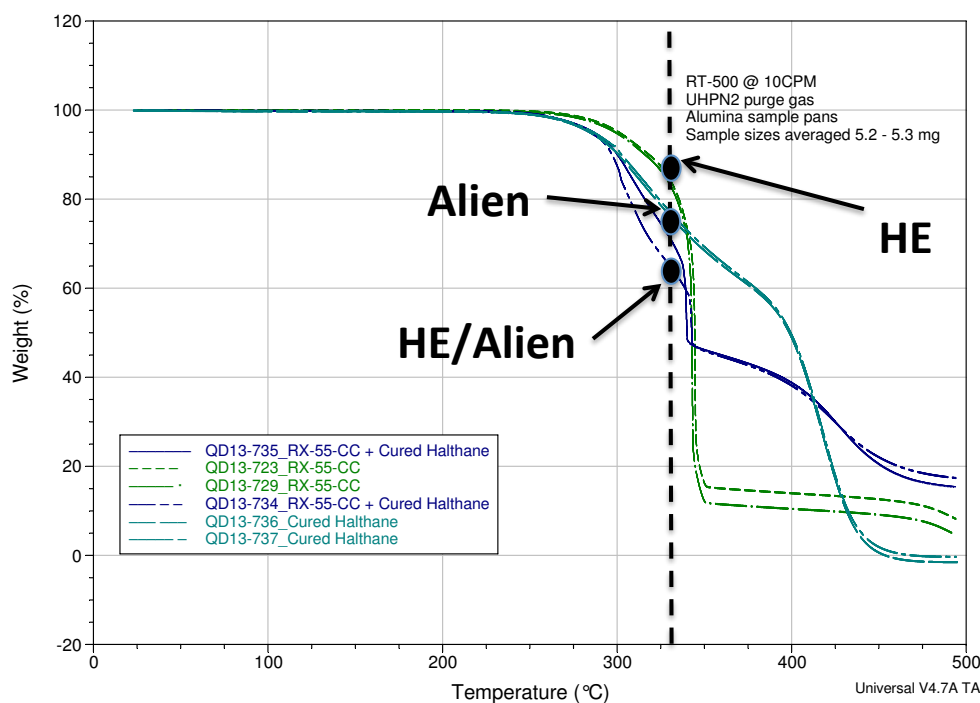
One will immediately recognize the two exotherms at 354°C and 384°C as those for the neat HE formulations themselves. This is a classic DSC case where there is not interaction between components and therefore is compatible.

In an attempt to correlate historical CRT compatibility data with that from other analytical techniques an identical campaign was started in FY2014 to study compatibility using thermal gravimetric analysis (TGA). The HE/material combinations studied in FY2014 by this approach are summarized below in Table 5. One will notice a concerted effort to study the same HE/materials pairs as was evaluated by CRT and DSC in Tables 3 and 4. This was done to allow the comparison of results from all three methods, which will enable both the pros and cons of all approaches to be more clearly evaluated. The TGA micro-compatibility results involving RX-55-CW are shown below in Table 5.

**Table 5.** Results from baseline TGA compatibility matrix for HE/materials combinations are shown below

HE/TGA	Lot#	RX-55-CC	RX-55-CW	TATB (new: DA)	TATB (new: WA)	LX-17-1 (legacy)	RX-03-HD (new LX-17-0)	RX-03-GX-2 (new LX-17-1)	LX-16	APC 2.5 (cured)	Halthane 73-18 (cured)	Halthane 88-3 (cured)	FK800 binder	SE1700 silicone
RX-55-CC	C-596	331												
RX-55-CW	C-596		339	31.30%			11.60%	5.3%	5.16%	4.06%		13.80%	4.28%	
TATB (new: DA)	C-666			NA										
TATB (new: WA)	C-668				NA									
LX-17-1 (legacy)	C-063					NA								
RX-03-HD (new LX-17-0)	C-666/FK800						NA							
RX-03-GX-2 (new LX-17-1)	C-668/FK800							NA						
LX-16	C-518								197					
APC 2.5 (cured)	#13-112									NA				
Halthane 73-18 (cured)											NA			
Halthane 88-3 (cured)												NA		
FK800 binder	#1												NA	
SE1700 silicone														NA
PASS														
BORDERLINE														
FAIL														
Not Completed														
Temperature used of														
Mass loss comparison														

To evaluate materials compatibility by TGA experiments are carried out in a nitrogen atmosphere. Separate TGA experiments are run on the individual pristine materials and then on a 1:1 weight mixture of them. The mass loss of the pristine materials is compared to that of the mixture of the HE and test material at a selected temperature. If the observed weight loss for the mixture is greater than the expected weight loss from the combination of the two materials (HE and alien) then it is an indication of an incompatibility. The larger the difference between the observed and expected weight losses the greater the degree of incompatibility. Material combinations displaying a weight difference of less than 4% ( $\% \text{ observed weight loss} - \% \text{ calculated (expected) weight loss}$ ) are considered compatible. A weight difference between 4% and 20% indicates a higher degree of incompatibility may exist and further testing is recommended. A weight difference of greater than 20% indicates an incompatibility between the two materials.. A representation of this type of data analysis is captured below in Figure 4.



**Figure 4.** Overlay of TGA curves for RX-55-CC, Halthane 88-3, and a 1:1 mass mixture of the two materials. The dashed line demotes the temperature at which the TGA compatibility data was analyzed. The black dots are the weight loss for the respective curves at that temperature.

Figure 4 contains an overlay of several different TGA runs of RX-55-CW, Halthane 88-3 and a mixture of the two materials. The dashed line represents the peak exotherm temperature for the neat RX-55-CW (~330°C). The weight loss values for each curve is determined from where the dashed line intersects each respective curve. While this approach has not been regularly utilized by the DOE, it is commonly employed by the other agencies (DoD and NATO) and is another metric for compatibility and is worth evaluation.

It is important to note that while the STANAG 4147 document calls for TGA analyses to occur at 2°C/minute the results presented in Table 5 are from analyses at 10°C/minute. As with the DSC method we wanted to explore the possibility of running at a higher ramp rate to reduce analysis time without affecting the data quality. Therefore a series of comparison runs at both scan rates were performed and analysis of the data shows some difference in the results. This result differs from our DSC study and indicates that the compatibility results from the TGA method are more sensitive than the DSC. This approach is very sensitive to the selection of the temperature for data analysis. This sensitivity is rooted in the steep drop in mass of the mixture at the HE decomposition temperature that is generally shown at ~350°C on the plot in Figure 4. The rapid change in weight loss with a very small change in temperature makes it critical that a representative value be used. Slight variation in the temperature selected can lead to dramatic differences in the PASS/FAIL criterion of the test. Since the higher heating rate of 10 CPM can lead to some thermal lag in material response (and more variability in temperature measurement)

it stands to reason that a more conservative approach to the test would necessitate the use of the 2 CPM method. This is an important outcome from our work this year and has enabled us to utilize the optimal conditions for a sound conservative evaluation of micro-compatibility using three analytical methods. Therefore while instructive for method development, the PASS/FAIL results in Table 5 will be re-analyzed at the 2 CPM value in future studies and their results updated when complete.

To summarize, this line of experimentation has attempted to display the differences between these three methods for determining compatibility and to some degree it has. Some trends were reinforced by the three methods while others were contradicted. If anything, this preliminary work has identified some material combinations of interest that will warrant more detailed study in more rigorous compatibility studies. While the results to date are interesting and worthwhile, it is our opinion that further study is needed to have more confidence in utilizing the TGA method in the suite of compatibility techniques. At least initially it appears that both the DSC and TGA methods are more sensitive than CRT to possible compatibility issues.

### ***Moisture Sorption/Diffusion Studies and Modeling***

In a warhead atmosphere it is important to quantify moisture ad/absorption, desorption, and diffusion through materials of interest. Moisture is prevalent as a latent species (i.e. not a degradation product) in many materials. It is almost impossible to completely remove moisture from a material via drying processes; therefore, it is one of the most likely species to outgas from many materials. Furthermore, moisture is an active species that is capable of degrading polymers. This FY such measurements and analysis was performed on LX-21. More specifically the outgassing speciation, and moisture sorption experiments were performed on LX-21 this year. The results from the moisture sorption experiments were used in diffusion and sorption modeling

The Intelligent Gravimetric Analysis of **sorption** (IGAsorp, designed and sold by Hiden Isochema) measures the weight change of samples in an environmentally controlled chamber. The vapor content and temperature in the chamber are controlled allowing for temperature dependent uptake and outgassing experiments. The chamber operates at ambient pressure, which is relevant to our applications, and uses large sample masses (up to 5g) so that both diffusion and sorption can be measured. A brief summary of the results of this line of investigation is reported here. A full description of the instrument and moisture experiments for LX-21 can be found in the ESC FY2014 Report titled “Development of Shared LLNL/SNL warhead atmosphere model” authored by Elizabeth Glascoe and Stephen Harley.

The outgassing speciation study showed no major outgassing species were observed (via the mass spectrometer) during the experiment, although a slow steady mass loss was observed. Most likely the sample was desorbing a small amount of moisture or other species, or there may have been some minimal sublimation. The sample was then exposed to 50% RH N<sub>2</sub> for 2 weeks at 80 °C. Again, no major species were observed in the mass spectrometer data, however, the slow steady mass loss continued. These experiments serve as a coarse analysis for moisture-induced chemistry. The absence of any major species indicates that moisture-induced degradation mechanisms in LX-21 are not a major reaction.

The moisture sorption experiments were performed at 30, 40, 50, and 60 °C. Results from these studies indicated a linear relationship between uptake mass and the square root of time, which is indicative of a diffusion process. In fact the experiments showed two regions of diffusion-limited uptake (one at low relative humidity (RH) and another at higher RH). One interpretation of these results would be that the two components of the formulation (Viton and LLM-105) have distinct and independent diffusion pathways. In order to de-convolute these mechanisms and properly model this material, each component must be experimentally measured and analyzed independently and future work will address this issue. The model also indicated that the Henry and Pooling modes are the dominant mechanisms for moisture uptake and Langmuir adsorption does not contribute to the sorption process.

### ***Long-term accelerated aging studies of LX-21***

The ultimate task for this program is to execute a long-term accelerated aging program for LX-21 and evaluate its aging properties under such conditions. The tasks described here help contribute to this goal. A more detailed aging plan was articulated recently in “Aging and Compatibility Plan for LX-21” (Authors: Gash and Glascoe). Part of that plan involved enacting gross LX-21 aging studies. Such studies will be relatively simple with confined powder and pressed parts of LX-21 in controlled atmosphere containers like that shown in Figure 5 below and aged for extended periods of time at our HE conditioning facility in B826 at Site 300.



**Figure 5.** Photo of an aging container for the LX-21 study. For reference the internal volume is approximately 100 mL.

During this FY, resources were expended to procure a number of these vessels as well as accompanying Swagelok valves. These vessels are well suited for our studies as all of their components are metal-based, from the 316 stainless steel base vessel to the copper knife-edge sealing gasket. This avoids the possible off-gassing contamination that can arise from polymer components. The vessel design has been pressure tested this year and is acceptable for our gross aging studies. In addition to studying neat LX-21 material and parts, we can do accelerated aging of simple multi-material assemblies. Procedure and work permits for this activity have progressed to the point of final review at the date of this report. These studies are set to begin in the first quarter of FY 15.

### 1.3 Conclusions

Several tasks in the LX-21 Compatibility effort were addressed in FY2014 and those include: 1) Select a mature formulation for future aging and compatibility experiments 2) Perform gross compatibility testing for a number of LX-21/material combinations, using three different evaluation techniques (DSC, TGA, and CRT) 3) Evaluate moisture sorption and diffusion characteristics of LX-21 4) Get the infrastructure and procedures in place to begin long-term accelerated aging experiments with LX-21.

The CRT, DSC baseline compatibility characterization has attempted to display the differences between these three methods for determining compatibility and to some degree it has. Some trends were reinforced by the three methods while others were contradicted. If anything, this preliminary work has identified some material combinations of interest that will warrant more detailed study in more rigorous compatibility studies. While the results to date appear interesting and worthwhile, it is our opinion that further study is needed to have more confidence in utilizing the TGA method in the suite of compatibility techniques. At least initially it appears that both the DSC and TGA methods are more sensitive than CRT to possible compatibility issues.

### 1.4 References

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